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(54) Title: EDIBLE PLASTIC SPREAD

### (57) Abstract

Edible plastic spread comprising a continuous fatphase and a dispersed aqueous phase, the fatphase comprising at least 5 % diglycerides and the glyceride fat of the fatphase having a N20 value of at least 3 wherein more than 70 % of the diglycerides are UU diglycerides and more than 45 % of the diglycerides are OO diglycerides wherein O indicates oleic acid residues and U indicates cis-unsaturated fatty acid residues with 18 carbon atoms. The incorporation of such diglycerides provides a more firm spread than would be obtained for a spread containing a typical triglyceride fatphase with the same overall fatty acid composition.

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### Edible plastic spread

The present application relates to edible plastic spreads comprising a continuous fatphase and a dispersed aqueous 5 phase, the fatphase comprising at least 5% diglycerides and the glyceride fat of the fatphase having an N20 value of at least 3.

Such spreads are known from WO 91/08677. This publication discloses margarine oil, and margarine made therefrom, that is low in trans fatty acids and low in "intermediate chain saturated fatty acids"  $(C_{\theta}-C_{16})$ . The margarine oil is produced by enzymatically interesterifying liquid oil, e.g. soyabean oil, with e.g. stearic acid, to obtain a non-15 random fatty acid distribution. The fatty acid distribution of the margarine oil should be within the following ranges:

			<pre>1, 3-glyceride positions (w%)</pre>	<pre>2 - glyceride position (w%)</pre>
20			posicions (wa)	posicion (we)
	C <sub>16</sub>		5 - 10	0 - 2.0
	C18		50 <b>- 7</b> 0	0 - 5.0
	C <sub>18:1</sub> cis		5 - 15	20 - 30
	$C_{18:2}^{18:1}$ all	cis	10 - 30	60 - 80
25	$C_{18:3}$ all		0 - 10	3 - 12

The diglycerides are formed as a byproduct of the enzymatic interesterification. No further information is given about the composition of the diglycerides.

EP 171 112 discloses the use of 5-30 wt% of diglycerides in fat to be used for making spreads to give products like margarine an elasticity, plasticity and melting behaviour more similar to those of butter. The fatty acid composition

- 35 of the diglycerides must meet the following requirements:
  - \* max 45% of saturated  $C_{16}$ - $C_{22}$
  - \* max 35% of saturated  $C_{12}$  and  $C_{14}$

30

- max 70% of mono-cis or trans and di-cis or trans unsaturated fatty acid residues with a chain length of 18 or more carbon atoms.
- 5 In the examples, diglycerides produced from rapeseed oil hardened to a melting point of 30°C are used.

EP 417 562 discloses that water-in-oil emulsions wherein the oil contains 0.1-80 wt% diglycerides, can be obtained with excellent properties in both stability and shape retentivity by incorporating 0.01-10 wt% based on the oil, of a specific phospholipid mixture. As diglyceride for example diglycerides derived from rapeseed oil, palm oil or lard can be used. Preferably the diglycerides contain at

15 least 70% unsaturated fatty acid residues.

EP 425 958 discloses that the emulsion stability, meltability in the mouth and shape retention of oil-in-water-in-oil emulsions can be improved by incorporating a 20 diglyceride having an open-tube melting point of below 20°C in the outermost oil phase and/or the innermost oil phase. The stability of the composition is further dramatically improved by using a phospholipid mixture as an emulsifying agent. Preferably the diglycerides contain 70% or more, especially 80% or more of unsaturated fatty acid residues. It is considered particularly desirable to use di-cis-unsaturated diglyceride at a content of 50% or more, especially 70% or more. By way of example, diglycerides

derived from rapeseed oil and maize oil are used.

JP 1,063,032 adresses the problem of stability of water-inoil emulsions with a high content of water. Such emulsions
suffer from oil-off, moisture release and phase inversion.
To obtain a stable emulsion, JP 1,063,032 describes to
include in the oil phase at least 10 wt% (w.r.t. the oil
phase) of a glycerol fatty acid diester having a melting

point below 30°C and to include saccharides in the water phase. The resulting emulsion has viscoelasticity which makes the emulsion stable. It is presumed that at the interface between oily and aqueous phases, an interaction 5 between the glycerol di-fatty acid ester and the sugars is generated whereby the surfacial membrane is stabilized giving an extremely stable emulsion. The ratio of oil to water phase is 50/50 to 1/99. In an example 1.5 kg of a dioleate product having a melting point of 12°C was 10 dissolved in 1.5 kg of soyabean oil to provide the oil phase. This was mixed with 7 kg of aqueous phase. As aqueous phase a 75% water solution of a branched oligosaccharide was used. The emulsion was prepared by mixing the composition for 5 minutes using a high shear 15 mixer. The emulsion was still stable after 1 month storage at 40°C. Similar results were obtained when using an oil phase consisting of a 1:1 mixture of soybean oil and diglycerides prepared by enzymic hydrolysis of soybean oil.

These emulsions are not intended as food products as such, but to be used in the manufacturing of food products. In view of the absence of solid glyceride fat at 20°C and the viscoelastic nature of the emulsion, these emulsions are not plastic spreads. Spreads are not normally stable at 40 °C, and such stability would not be desirable in view of the organoleptic characteristics desired for plastic spreads.

JP 1,137,938 adresses the same problem as JP 1,063,032. The solution now proposed is the incorporation of at least 20 wt% (w.r.t. the oil phase) of glycerol-di-fatty acid ester having a rising melting point of at most 30°C and the use of at least 2 wt% (w.r.t. the total composition) of an electrolyte. The ratio of oil to waterphase is from 75/25 to 5/95. In this case the viscoelasticity giving the high stability is presumed to be related to an interaction

between the diglycerides and the electrolyte at the surfacial membrane. In an example 2.5 kg glycerin dioleate having a melting point of 12 °C is used as oil phase. 25 parts of the dioleate, and an aqueous phase of 73 parts of water and 2 parts of NaCl are mixed for 5 minutes in a high shear mixer to produce the emulsion. The emulsion remains stable when stored at 30°C for 1 month. Other examples include the use of diglyceride produced from soyabean oil fatty acids, which gives similar results.

10

These emulsions also are not plastic spreads.

For health reasons certain customers find it desirable to reduce the content of saturated fatty acids, particularly

- 15 C<sub>12</sub>, C<sub>14</sub> and C<sub>16</sub> fatty acids, and of unsaturated fatty acids that contain one or more trans double bonds. For making edible plastic spreads such as butter, margarine and reduced fat spreads, typically triglycerides with 2 or 3 saturated or trans-unsaturated fatty acid residues are
- 20 included to be able to obtain the desired plastic product structure.

For a good spread, especially the firmness and plasticity in the temperature range of 5-10°C, up to 15°C is

- 25 important. Todays customer stores spreads in the refrigerator and expects the product to be ready for use immediately after taking it therefrom. Yet, the product may be left for some time outside the refrigerator, e.g. at the breakfast table, and warm up to ambient temperature. The
- 30 spread must be able to withstand this and still be plastic and spreadable. To achieve this the glyceride fat of the fatphase of the spread should contain a sufficient amount of solid fat at 20°C. This can suitably be expressed by the N value, indicating the amount of solid fat as measured by
- 35 NMR. For good results the N20 of the glyceride fat of the fat phase should be at least 3.

It is an object of the present invention to provide plastic spreads with an improved balance of product firmness and the nutritional properties of the fatty acid residue composition. The plasticity, spreadability, oral melt and other sensoric customer perceivable product properties should not be adversely affected.

This is achieved to a considerable degree by the present invention. The invention provides an edible plastic spread comprising a continuous fatphase and a dispersed aqueous phase, the fatphase comprising at least 5% diglycerides and the glyceride fat of the fatphase having an N20 value of at least 3, wherein more than 70% of the diglycerides are UU diglycerides and more than 45% of the diglycerides are OO diglycerides. O indicates oleic acid residues and U indicates cis-unsaturated fatty acid residues with 18 carbon atoms. Thus, U comprises oleic (cis), linoleic (cis, cis) and linolenic (cis, cis, cis) acid residues.

20 Preferred embodiments of the invention are given in claims 2-7.

In typical spreads based on fats consisting essentially of triglycerides, all-cis unsaturated fatty acid residues do
25 not contribute to the firmness of the product. The structure of such products is largely provided by saturated and trans-unsaturated fatty acids, especially by the triglycerides containing 2 or 3 of those fatty acids. It was now found that glycerides not containing such fatty
30 acids can contribute to product firmness especially at 5-10°C, provided that the glyceride is a diglyceride, particularly 00 being effective. Accordingly, by using 00 in the fat to be used for making the spread, a higher product firmness can be achieved than would result from a triglyceride fat with the same overall fatty acid composition. Reversely, the same product firmness can be

realised with a composition containing less saturated and trans containing unsaturated fatty acid residues, compared with a conventional triglyceride fat composition.

- 5 It is known that the firmness of spreads can be increased by adapting the processing without changing the product composition. However, such a way to increase the firmness is accompanied by a change in product texture. The product becomes less plastic and more brittle, which is not
- 10 appreciated by customers. With the present invention, the firmness can be increased without adversely affecting the nutritional fatty acid profile and without any adverse effect on the customer perceivable product properties. A suitable method to quantify customer perceivable texture
- 15 properties is Texture Profile Analysis (TPA) as described in Food Technology, July 1978, 62-66.

The invention is particularly advantageous for relatively firm spreads, e.g. spreads that are usually packed in

- 20 wrappers, but it can also be used in softer spreads, e.g. tub products. Accordingly, the present spread preferably has a Stevens 4.4 value at 10°C of 25 600, more preferably 75 500. The Stevens 4.4 value at x °C is an indication of the firmness of the product at x °C. It is
- 25 measured using a 4.4 mm φ cylinder in a Stevens LFRA

  Texture analyzer (ex Stevens Advanced Weighing Systems,

  Dunmore, UK), loadrange 1000 g. operated "normal" and set
  at 10 mm penetration depth and 2.0 mm/s penetration rate.
- 30 The invention can be used for any edible plastic spread having a continuous fatphase and a dispersed aqueous phase, but the advantages are more pronounced in products for which the firmness obtained is strongly dependent on the fat used in the fatphase. Accordingly, the spread
- 35 preferably comprises 20-90% of fatphase and 80-10% of aqueous phase, more preferably 35-85% of fatphase and 65-

15% of aqueous phase, 70-85% of fatphase and 30-15% of aqueous phase being particularly preferred.

The fatphase consists largely of di- and triglyceride fat.

5 In addition, the fatphase may comprise small amounts of other components e.g. colorant, flavour, emulsifier and the like. Specifically, the fatphase may contain some monoglyceride which acts as emulsifier, but preferably the amount of monoglyceride in the fatphase is low, typically 10 below 3%, preferably less than 1%.

The aqueous phase of the spread has a composition as is commonly used in spreads. It contains water and may further include other components such as milk or ingredients

15 derived from milk, flavours, gelling and structuring agents e.g. starches, salt, etc. The presence of excessive amounts of saccharides, e.g. sucrose, and of electrolytes is not preferred because they will impart taste and flavour to the spread that the customer does not expect. The amount of

20 saccharide in the aqueous phase is preferably less than 30%, more preferably less than 20%, especially less than 10%. It is most preferred not to include any saccharide as such in the aqueous phase at all. The product preferably contains less than 2% electrolytes, more preferably less than 1.5%, especially less than 1%.

The fatphase preferably comprises 10-60% diglycerides, more preferably 10-40% diglycerides. Of these diglycerides preferably 50-100%, more preferably 60-85% are OO diglycerides. Apart from such minor components as described above, the remainder of the fatphase preferably consists of triglycerides. The OO diglycerides contribute to the firmness of the product at temperatures where it counts most, e.g. 5-15°C, especially 5-10°C. Yet the customer expects from a spread that it gives a cool impression in the mouth upon eating it. This cool impression of spreads

is caused by the melting of solid fat upon the temperature increase of the spread in the mouth. To achieve this coolness sufficient solid fat should be present in the glyceride fat at 20°C and preferably also still at 30°C. At 5 body temperature, after a little while preferably little or no glyceride fat should remain in solid form. Accordingly the glyceride fat of the fatphase preferably has N20 of 3-35, more preferably of 7-30, especially 9-25. N30 preferably is 0.5-15, more preferably 1-10, especially 3-8. 10 N35 preferably is 0-7, more preferably 0-5 especially 0-3. At temperatures of 20-30°C the solid fat of the glyceride fat essentially originates from triglycerides. Therefore as part of the glyceride fat, suitable triglycerides should be incorporated to achieve this desired coolness. As 15 triglycerides any oils or fats may be used as are commonly employed for making plastic spreads. Oils or fats from animal or vegetable origin may be used, they may have been modified by way of fractionation, interesterification and/or hydrogenation, and blends of such oils and fats can 20 be used. Preferably oils or fats from vegetable origin are employed. Preferably, for nutritional reasons, oils and fats rich in  $C_{12}$ - $C_{16}$  fatty acids or trans unsaturated fatty acids are avoided or are used only in small amounts. If fats rich in saturated fatty acids are employed, preferably 25 the saturated fatty acid predominantly consists of stearic acid  $(C_{18})$ . Accordingly, of the saturated fatty acid residues in the fatphase, preferably at least 50%, more preferably 70-100% are stearic acid residues, a content of  $C_{18}$  of 80-95% of the saturated fatty acid residues being 30 particularly preferred. The content of trans fatty acid residues in the fatphase is preferably less than 15%, more

To obtain optimal contribution of 00 diglycerides to the 35 firmness of the product, preferably a fatphase with

preferably less than 7%, especially less than 3%.

relatively little complexity is used. This can for example be achieved by using only low contents of fatty acid residues with a chain length different from 18. A fatphase with a fatty acid composition comprising at least 75%, more preferably 80-100%, especially 85-97% stearic acid and (all) cis unsaturated fatty acid with chain length 18 is preferred. The presence of a very small amount, e.g. 2-5%, of other fatty acids such as palmitic acid may be beneficial for the structure of the product.

10

The diglycerides to be incorporated in the product can be prepared by conventional means, e.g. by means of hydrolysis of triglycerides or esterification of glycerol. Chemical or enzymatic reaction methods can be used. The resulting

- 15 mixture will typically contain triglycerides, diglycerides and monoglycerides. It can, after refining, be used as such, or it can be modified e.g. to reduce the monoglyceride content, e.g. by means of distillation. A preferred method for producing the diglycerides is by use
- 20 of enzymatic rearrangement of an appropriate triglyceride oil with glycerol using lipase. For the present invention it is not important whether the diglycerides are 1,2 diglycerides or 1,3 diglycerides. Both can be used as well as combinations of 1,2 and 1,3 diglycerides. Preferred oils
- 25 for use as starting material for the production of the diglycerides are oils containing at least 85% of U fatty acid residues and at least 70% of O fatty acid residues. Contents of 90-98% U and of 75-90% O are preferred. For example, varieties of safflower, and especially of
- 30 sunflower and rapeseed oil with high oleic acid contents are particularly suitable.

Throughout this specification, all parts and percentages are parts and percentages by weight, unless otherwise

35 indicated. Unless specified otherwise, N-values are measured after the following stabilisation: heating to

80°C, at least 5 minutes at 60°C, 60 minutes at 0°C, 30 minutes at measuring temperature. Diglyceride contents are measured with GLC. The expressions "comprises" and "comprising" include "consists of" and "consisting of" except where indicated otherwise.

The accompanying figures show force-time diagrams obtained in TPA analyses as is further described in the examples.

### 10 EXAMPLE 1

A diglyceride concentrate was prepared by interesterifying a mixture of 92.5% high oleic sunflower oil with 7.5% glycerol at 220°C using NaOH as catalyst. The resulting mixture was separated by short path distillation to reduce 15 the monoglyceride content.

The resulting mixture contained 51.3% triglycerides, 47.3% diglycerides and 1.4% monoglycerides. The fatty acid composition was:

20

25

Palmitic	4.1	°
Stearic	4.4	%
Oleic (cis)	82.6	% જ
Linoleic (cis,	cis) 7.6	%
Others	1.3	%

This was the same as the fatty acid composition of the high oleic sunflower oil used as starting material.

30 The diglyceride composition was approximately:

	00	66	왕
	SS	1	왕
	so	17	÷
	SL	2	옿
35	OL	13	ş
	LL	1	ક

wherein S indicates saturated fatty acid residues and L indicates linoleic (cis, cis) and linolenic (cis, cis, cis) acid residues.

A fat blend was prepared with the following composition:

- 60% hardstock (no trans fatty acid residues)
- 30% of the above described diglyceride concentrate
- 10% high oleic sunflower oil

5

The hard stock had the following triglyceride composition:

	4.4 %	SSS
	46.5 %	SUS
	2.3 %	SSU
10	16.0 %	<b>S</b> 00
	30.8 %	lower melting triglycerides, e.g. USU, SUU other than SOO, and mainly UUU.

15 The fatty acid composition of the hardstock and of the overall fatblend were:

	Fatty acid	Hardstock	<u>Fatblend</u>
	Palmitic	3.6 %	3.8 %
	Stearic	43.9 %	28.1 %
20	Oleic	32.4 %	52.5 %
	Linoleic	18.5 %	14.1 %
	Others	1.6 %	1.5 %

The fatblend contained 14.2 % diglycerides.

25

The N-values were measured using both the normal stabilisation ("short") of heating to 80°C, at least 5 minutes at 60°C, 60 minutes at 0°C and 30 minutes at the measuring temperature, and a "long" stabilisation of

30 heating to 80°C, at least 5 minutes at 60°C, 16 hours at 0°C and 30 minutes at measuring temperature.

The results were:

		"Short"	"Long"
35	<b>N</b> 5	31.9	42.9
	N10	28.7	31.8
	N20	17.8	18.9
	N30	4.3	4.4
	N35	3.5	3.2

To this fatblend 0.1% lecithin was added. An aqueous phase was prepared by solving in water 1% skimmilk powder and citric acid to obtain a pH of 4.4. 17% of aqueous phase was mixed with 83% fatphase at 60°C. The mixture was rapidly chilled by passing it through lab scale spread equipment comprising 2 surface scraped heat exchangers. The temperature of the product leaving the equipment was 10°C. The product was stored at 10°C.

10 For comparison the experiment was repeated, except that in the fatblend instead of the 30% diglyceride concentrate 30% high oleic sunflower oil was used.

The	N-v:	alues	were:	•

15		"Short"	"Long"
	<b>N</b> 5	33.6	43.3
	N10	30.0	34.9
	N20	18.4	17.1
	N30	5.2	4.6
20	N35	2.5	3.1

These N-values are rather similar to those of the example fatblend. If anything, the solid fat contents of the comparative blend were somewhat higher.

25

The Stevens 4.4 values of the product 35 min. after production and after 1 and 4 weeks storage were:

		Temp.	Example	<u>Comparison</u>
	after 35 min.	19°C	299	45
30	after 1 week	10°C	591	239
	after 4 weeks	10°C	590	280

The product containing the diglycerides was much firmer than the comparative product even though it contained the same hardstock, had the same overall fatty acid composition and had very similar solid fat contents.

### Comparative example

A diglyceride concentrate was prepared in a manner similar as described above but rapeseed oil was used instead of high oleic sunflower oil.

5

The diglyceride concentrate contained 33.5% triglycerides, 66% diglycerides and 0.5% monoglycerides. It had the following fatty acid composition.

	Palmitic	4.7 %
10	Stearic	1.5 %
10	Oleic (cis)	58.3 %
	Linoleic (cis, cis)	22.8 %
	Linoleic (cis, cis, cis)	9.4 %
	Others	3.3 %

15 This fatty acid composition is very similar to that of the rapeseed oil starting material.

The diglyceride composition was approximately:

	00	35	કૃ
20	SS	1	%
	SO	9	ફ
	SL	5	ક
	OL	40	%
	LL	10	%

25

The above example was repeated except that as fatblend was used:

A 60% hardstock (as in above example)

40% of the diglyceride concentrate derived from rapeseed oil.

30 B 60% hardstock

40% rapeseed oil

The diglyceride content of fatblend A was 26.4 %, that of fatblend B was 1.5%.

35 The Stevens 4.4 values of the products were:

	Temp.	Sample A	Sample B
after 35 min.	20°C	28	48
after 1 week	10°C	93	151
after 4 weeks	10°C	146	154

Sample A with the diglycerides derived from rapeseed oil was not more firm than sample B which had the same overall fatty acid composition but did not contain significant amounts of diglycerides.

The benefit achieved in the example with diglycerides with a high OO content (66%) was not achieved when the diglycerides contained only 35% OO, even though in the comparative example a higher diglyceride content was used in the fatblend.

### Example 2

A diglyceride concentrate was prepared as described in example 1 except that a different batch of high oleic

15 sunflower oil was used. The concentrate contained 69% diglycerides. The fatty acid and diglyceride composition were:

		%		%
	C16:0	4.2	00	63
20	C16:1	0.1	SS	1
	C18:0	4.5	so	16
	C18:1	79.6	SL	2
	C18:2	9.7	OL	16
	C20:0	0.4	LL	1
25	C20:1	0.3	Othe	r 1
	C22:0	0.9		
	C24:0	0.2		

With this concentrate the following fatblend was prepared:

- 30 44 parts diglyceride concentrate
  - 30 parts palm oil
    - 6 parts palmkernel oil
  - 20 parts of a randomised mixture of 57% fully hardened palmkernel oleine and 43% fully hardened palm oil.

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For comparison, the same fatblend was prepared except that instead of the diglyceride concentrate, high oleic sunflower oil was used.

The fatty acid composition of the two fatblends was (determined by GLC):

			Ex.2	Comp.2
10	C8:0		0.7	0.7
	C10:0		0.7	0.6
	C12:0		8.4	8.2
	C14:0		3.2	3.1
	C16:0		21.0	20.7
15	C16:1	cis	0.1	0.1
	C17:0		0.1	0.1
	C18:0		10.5	10.4
	C18:1	cis	46.7	47.0
	C18:2	cis cis	7.4	7.9
20	C18:3	all cis	0.1	0.1
	C20:0		0.3	0.3
	C20:1	cis	0.2	0.2
	C22:0		0.4	0.4
	C24:0		0.1	0.1

25

Within the accuracy of the analysis, the fatty acid composition of the 2 fatblends is the same.

The solid fat contents of these fatblends were:

5		Short stabil	lisation Comp.2	Long stab Ex.2	ilisation Comp.2
10	N5	41.1	39.0	48.7	42.0
	N10	35.3	33.9	39.4	36.5
	N15	25.8	25.3	28.4	28.5
	N20	19.2	18.9	19.6	19.7
	N30	7.8	7.5	9.1	8.5
	N35	4.0	4.2	3.7	4.1

15

20

These data indicate:

- \* At low temperatures the example fatblend has higher solid fat contents than the comparative fatblend: at higher temperature they are the same (within the accuracy of the measurement).
- \* It appears that both fatblends are not likely to be fast crystallising.
- 25 Using these fatblends, fatphases and also aqueous phases were prepared:

	Fatphase:	79.75	parts	fatblend
		0.05	parts	monoglyceride
		0.05	parts	lecithin
30		0.15	parts	${\tt B}$ - carotene solution
				_
	Subtotal	80.00	parts	
	Aqueous phase:	18.90	parts	water
35		0.70	parts	wheypowder
		0.30	parts	salt
		0.10	parts	potassium sorbate
		p.m.		citric acid to pH 4.8
				· ·
40	Subtotal	20.00	parts	

From the fatphase and aqueous phase a margarine premix was prepared which was kept at 60°C.

The premix was passed through a small scale Votator unit 5 with ACAB sequence wherein

A is a scraped surface heat exchanger.

The first A-unit was operated at 1000 rpm.

The second A-unit was operated at 800 rpm.

C is a stirred crystalliser operating at 300 rpm.

B is a resting tube.

The cooling on the A-units was such that the composition after the second A-unit had a temperature of about 15-16°C. Two series of trials were done, one with a throughput of 15 3.4 kg/h, the other with 5.6 kg/h.

The product leaving the B-unit was packed in wrappers. The products were stored at 15°C.

- 20 After 1 week an initial assessment was made. This showed that both the trial and the comparative product produced at a throughput of 3.4 kg/h were quite good products. Both products produced at 5.6 kg/h however were rather brittle. This confirmed the impression from the solid fat content
- 25 measurements that the fatblends were not fast crystallising. It further indicated that the use of the diglyceride concentrate compared with the high oleic sunflower oil in the blend, did not cause substantial changes in the crystallisation properties of the fatblend.

Of the products produced at 3.4 kg/h the firmness was assessed using Stevens 4.4 values after 1 and 2 weeks storage and the products were evaluated blind by an experienced panel after 2 weeks storage.

30

10

		1 week storage Ex.2	Comp.2	2 weeks s Ex.2	torage Comp.2
St St St St	15	876 379 196 89	568 243 118 104	- 401 203 96	250 175 121

10 These data show that the product of the example is substantially more firm than the comparative example at the lower temperatures, more so than anticipated based on the solid fat contents of the fatblends. They further show that only little posthardening occurred which confirmed that the processing applied had been appropriate in view of the crystallisation characteristics of the fatblends.

The panel easily recognised the difference in firmness between the products when taken from the refrigerator.

20 Apart from that they judged the two products to be equally good. Specifically both products had very good plasticity and very good melt down in the mouth.

### Example 3

25 Using the same diglyceride concentrate as in example 2, fatblends were prepared as follows:

	EX.3	Comp.3
Hardstock of example 1	60	60
Diglyceride concentrate	40	-
30 High oleic sunflower oil	-	40

Analysis showed that the diglyceride content of the example fatblend was 29% while that of the comparative fatblend was 2%.

35

The fatty acid composition and the solid fat content measurements of the fatblends gave the following results.

	Ex.3	Comp.3
C14:0	0.1	0.1
C16:0	3.9	4.0
C16:1 cis	0.1	0.1
C17:0	0.1	0.1
C18:0	28.6	28.3
C18:1 cis	50.5	50.7
C18:2 cis cis	15.2	15.2
C18:3 all cis	0.1	0.1
C20:0	0.7	0.7
C20:1 cis	0.2	0.2
C22:0	0.5	0.5
C24:0	0.2	0.1

		Short s Ex.3	tabilisation Comp.3	Long Ex.3	stabilisation Comp.3	
20	N 5	36.2	33.5	47.1	43.4	
	N10	28.5	27.1	33.7	34.5	H
	N15	22.5	24.5	24.8	25.6	- 1
	N20	17.5	18.4	19.2	18.9	I
	N30	3.9	4.2	4.2	4.9	- 1
25	N35	3 1	2 7	2.6	3 8	

These data indicate:

- \* Within experimental accuracy, the fatblends have the same fatty acid composition.
- 30 \* Except at 5°C the solid fat contents of the two blends are similar. At 5°C the example fatblend has higher solid fat contents.
  - \* It appears that both fatblends are not likely to be fast crystallising.

Using these fatblends, premixes were prepared from fatphases and aqueous phase as described in example 2.

Margarine was prepared in 2 series of trials. In one trial 40 the Votator sequence was ACAC:

First A-unit: 1000 rpm First C-unit: 100 rpm

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800 rpm. Composition temperature after Second A-unit:

second A-unit : 9-10°C

50 rpm. Second C-unit:

5 In the second trial, an AA sequence was applied

First A-unit : 1000 rpm

Second A-unit: 1000 rpm. Composition temperature

ex. A2 : 9-10°C

10 The throughput in all cases was 3.4 kg/h.

The products were filled in tubs and stored at 15°C.

Firmness measurements after 1 and 2 weeks gave the

15 following results:

15 1	ollowing results:				
		After Ex.3	1 week Comp.3	After 2 Ex.3	weeks Comp.3
	Process ACAC				
20	St 5 St 10 St 15 St 20	1289 821 564 229	721 603 540 247	- 897 445 211	514 511 210
	Process AA				
25	St 5 St 10 St 15 St 20	686 447 291 110	505 355 308 129	- 478 301 99	385 321 123

These data show that the products of the example were substantially more firm at 5 and 10°C than the comparative 30 products. At higher temperatures they were nearly the same considering the accuracy of the measurement. Little or no posthardening occurred.

For these samples TPA measurements were carried out using 35 the same equipment as described above for Stevens value measurement.

Probe 4.4 mm ø

Speed 2.0 mm/s

Penetration depth 10.0 mm

The probe is pushed into the sample, pulled out, pushed in 5 again and pulled out again. The required force is recorded versus time. The force-time plots obtained are shown in Figures 1-4:

Figure 1: Ex.3 ACAC process

Figure 2: Comp.3 ACAC process

10 Figure 3: Ex.3 AA process

Figure 4: Comp.3 AA process

The shape of the plots for figures 1 and 2 are very similar and so are those of figures 3 and 4, showing that these pairs of product are very similar in terms of plasticity.

15

As the probe is pushed into the product for the first time, immediately a substantial force is required. This required force gradually increases during the first 5 seconds until the probe reaches its maximum depth. In figures 3 and 4,

- 20 the slope of this first part of the curve is nearly smooth, indicating a homogeneous product. This part of the curve in figure 1 and 2 is however very irregular, showing that these products are inhomogeneous, i.e. brittle.
- 25 Then the probe is slowly pulled out of the product. For this a force in the opposite direction is needed. In figures 1 and 2, after a short while the required force to pull out the probe becomes very small. The bottom surface of the probe has released itself from the product and
- 30 little force is needed to overcome the adherence from the side contacts of the probe and the product.

In figures 3 and 4, however, as the probe is being pulled out, substantial force continues to be required. The 35 product quickly recovers from the intrusion and subsequent

pulling out of the product, indicating good plasticity of the product.

When after 10 seconds, the probe moves again into the product, in figures 1 and 2, initially little force is required. The first movement cycle of the probe had left a little channel and therefore in its second cycle the probe meets with little resistance. In figures 3 and 4 however, when the probe begins to move into the product, immediately substantial force is required, indicating that the product structure had already largely recovered from the first movement cycle of the probe.

Upon the second time pulling out of the probe, between

15 figures 1 and 2 on the one hand, and 3 and 4 on the other
hand, a similar pattern occurs as for the first time the
probe was pulled out.

From such graphs, in particular 2 quantitative measures can 20 be derived which characterize the plasticity of the product: the springiness and the cohesiveness.

Springiness is that part of the time during which the probe is pushed into the product for the second time, that

25 substantial force is required. Cohesiveness is the ratio of the second positive surface area (force x time during 2nd time pushing in of probe) and the first positive surface area (force x time during 1st time pushing in of probe) of the force time plot. For both parameters holds that the

30 larger the ratio (both are theoretically at most 1) the better the plasticity of the product.

The following results were obtained:

	ACAC process Ex.3 Comp.3		AA process Ex.3 Comp.3	
Springiness	0.3	0.5	1.0	1.0
Cohesiveness		0.2	0.5	0.5

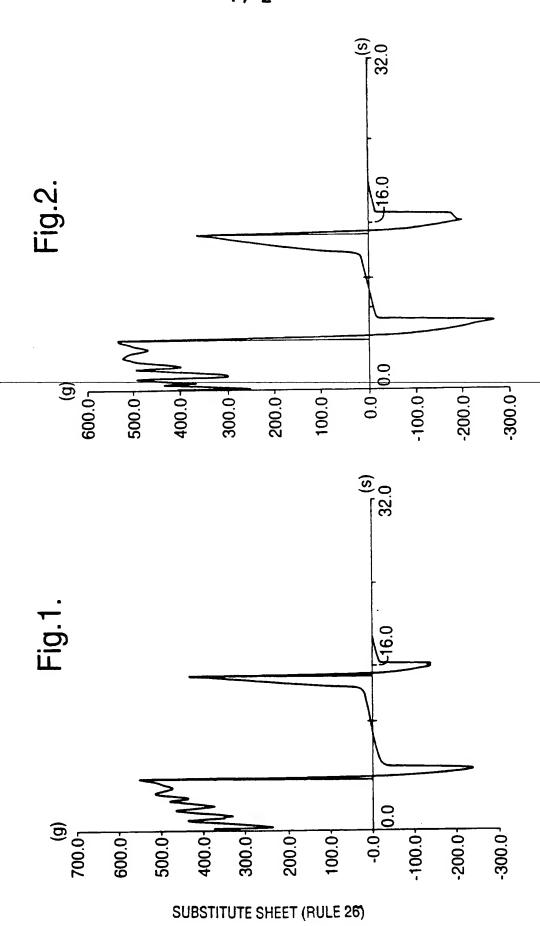
This shows that the products from the AA process had equally good plasticity and were much better than the products of the ACAC process. As was shown above, the products of the ACAC process were more firm than those of the AA process. This illustrates the above comment that if products are made to be more firm by changing the processing without changing the composition, the plasticity of the product is adversely affected. With the fatblend of the examples without change in the fatty acid composition, more firm product can be obtained without negative influence on the plasticity.

The products were also evaluated by an experienced panel. The higher firmness of the products of the example compared 20 with the comparative products, when taken from the refrigerator, was easily noticed. Otherwise the products from the ACAC process were found to be very similar. Both had acceptable melt down in the mouth. For both spreadability was not good. The two products from the AA 25 process both had very good melt down in the mouth and both had very good spreadability. The product of the example was even found to be slightly better than the comparative product in terms of plasticity. Overall, this product (Ex.3 AA process) was found to be the best of all products of examples 2 and 3.

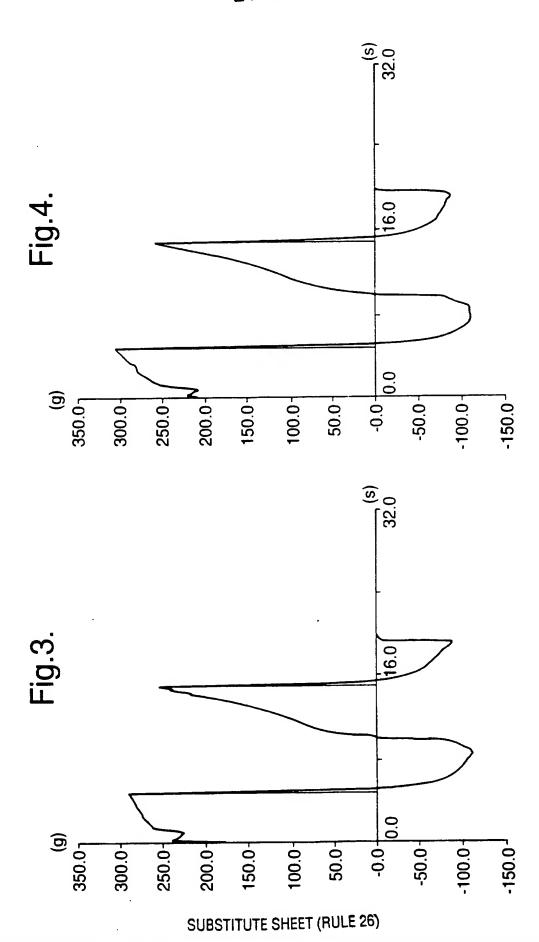
#### CLAIMS

- 1. Edible plastic spread comprising a continuous fatphase and a dispersed aqueous phase, the fatphase comprising at least 5% diglycerides and the glyceride fat of the fatphase having a N20 value of at least 3 characterised in that more than 70% of the diglycerides are UU diglycerides and more than 45% of the diglycerides are OO diglycerides wherein O indicates oleic acid residues and U indicates cisunsaturated fatty acid residues with 18 carbon atoms.
- 2. Spread according to claim 1, wherein 75-100%, preferably 80-90% of the diglycerides are UU diglycerides.
- 3. Spread according to claim 1 or claim 2, wherein 50-100%, preferably 60-85% of the diglycerides are OO diglycerides.
- 4. Spread according to any one of claims 1-3, wherein the fatphase comprises 10-60%, preferably 10-40% diglycerides.
- 5. Spread according to any one of claims 1-4, wherein of the saturated fatty acid residues of the glyceride fat, at least 50%, preferably 70-100%, more preferably 80-95%, are stearic acid residues.
- 6. Spread according to any one of claims 1-5 wherein of the fatty acid residues of the glyceride fat, at least 75%, preferably 80-100%, more preferably 85-97% are fatty acid residues with 18 carbon atoms.

7. Spread according to any one of claims 1-6, comprising 20-90% of fatphase and 80-10% of aqueous phase, preferably 35-85% of fatphase and 65-15% of aqueous phase, more preferably 70-85% of fatphase and 30-15% of aqueous phase.



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tional Application No PCT/EP 96/01323

A.	CLASSI	FICATION	OF	SUBJECT	MATTER
П	PC 6	A23D7	70	Α	

According to International Patent Classification (IPC) or to both national classification and IPC

### **B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 A23D C11C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Further documents are listed in the continuation of box C.	Patent family members are listed in annex.
* Special categories of cited documents:  'A' document defining the general state of the art which is not considered to be of particular relevance.  'E' earlier document but published on or after the international filing date.  'L' document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified).  'O' document referring to an oral disclosure, use, exhibition or other means.  'P' document published prior to the international filing date but later than the priority date claimed.	'T' later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention.  'X' document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone.  'Y' document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.  '&' document member of the same patent family
Date of the actual completion of the international search  1 July 1996	Date of mailing of the international search report  16.07.96
Name and mailing address of the ISA  European Patent Office, P.B. 5818 Patentiaan 2  NL - 2280 HV Rijswijk  Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  Fax: (+31-70) 340-3016	Authorized officer  Dekeirel, M

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT  Relevant to claim N				
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